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CASTABLE ILLUMINATION FLARES
by

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~~Propulsion Development Department~~

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SUMMARY

Two oxygen enriched binder systems were used to prepare caat illumination flares. An inorganic salt, magnesium perchlorate, was dissolved in a polymerizable binder system of mixed butylacrylate and acrylonitrile. The luminosity obtained in castings (4.25 inch diameter) approached an average intensity of ~~2×10^6 cp.~~ 2 million cp.

The second system utilized triethylene glycoldinitrate in a binder system of Dynamar HX 736, a prepolymer with a 30% oxygen content, with Dynamar HX 740 curing agent. Limited data indicate that a luminosity intensity of ~~2×10^6 cp~~ is attainable.
2 million

When 7.25 inch diameter candles of both types were made fragmentation occurred during static firing and large pieces were ejected from the flare case. ~~Work was discontinued for lack of funds.~~ The maximum practical diameter and optimum performance have not been determined.

CASTABLE ILLUMINATION FLARES

BACKGROUND

Work was initiated in 1963 on the possibility of developing a castable flare composition. This work was supported by NAVAIRSYSCOM under the Pyrotechnics Supporting Research Work Unit of the Air Launched Pyrotechnics Exploratory Development Program. Technical Coordinator and Project Administrator was Mr. Ray Szypulski. The problem as originally stated was to develop a castable composition suitable for illumination flares so that the cost of fabrication would be decreased from that of the then used pressed magnesium, sodium nitrate, Laminac. It was considered OK under the ground rules for the developed formulation to be less efficient than the pressed mixes provided safety could be increased. Later these rules were revised to require that the mixes also be competitive on a luminance efficiency basis to the pressed candles.

It was felt from the beginning that to obtain a castable flare formulation of moderate to good performance it would be necessary to reduce the amount of hydrocarbon diluent as much as possible. It was for this reason that monomer soluble salts were investigated. That is not so much to increase the oxidizer content of the binder, but to decrease the actual amount of hydrocarbon present. While the two ways of looking at the problem may give the same result, the thinking behind the work was different than the work pursued by Dow Chemical for the Army and then the Air Force, or the work on tamper castables by Thiokol. The work is very similar to that carried out later at NAD, Crane where strontium perchlorate was used dissolved in an diglycidyl acrylate for a binder in red flare mixes. This work interestingly enough was initiated independently of the work at China Lake. Fortunately, there were no fire or explosive incidents with the magnesium perchlorate with the chosen monomers, although Waite, (Ref. 1) reported such difficulties with related acrylates.

In preliminary work not reported in this paper, one of us (GH) experimentally evaluated the solubilities of various oxidizers and perchlorates in a variety of commercially available polymers. The particular basic components, magnesium perchlorate, acrylonitrile, and butylacrylate were chosen because of the relatively low cost of the organic components, the fact that ANBA polymers in general have good physical properties, that perchlorates were known to catalyze the polymerization when present in small amounts, and that there were the fewest new elements injected into the basic illumination flare reactants so that the luminance would not suffer degradation from siphoning available chemical energy into new emission bands.

Early in the program it was necessary to make a brief study of the polymerization reaction of the ANBA system in the presence of magnesium perchlorate (MgP). When normal amounts of free radical catalysts were used, the polymerization reaction tended to run away with strong foaming and some decomposition. It was only by decreasing the amount of free radical catalyst that successful cures were obtained. Alternatively it did prove possible to carry out the polymerization of the pure binder system without the free radical catalyst in the presence of sunlight, and such a technique was used when studying the physical properties of the neat binder system.

Due to the well known water absorption properties of magnesium perchlorate (sold under the trade name Dessichlora) some preliminary experiments were conducted on the water absorption characteristics of the neat binder system. The system was prepared without the free radical catalyst within a glass vial. Upon removal from the glass vial it was weighed on an analytical balance and stored in the laboratory and exposed to the ambient air. A gradual increase in weight occurred over a period of several days during which the temperature and relative humidity were kept fairly constant by air-conditioning to $74 \pm 3^{\circ}\text{F}$ and 40% RH. Then during a subsequent air conditioning failure and increased humidity, 92°F and 70% RH the sample gained approximately 5% in weight, but more significantly became very rubbery. It also appeared that water had only penetrated approximately 20% of the way in and the center was still hard and clear. This led to the later investigation of including a third material as a plasticizer.

MAGNESIUM PERCHLORATE ENRICHED BINDER SYSTEM

Butylacrylate and acrylonitrile polymerize to form an effective binder and both substances dissolve magnesium perchlorate to a substantial amount. This system (MgANBA) was used in preparing castable mixes for illumination flares (Ref. 2 and 3).

Solubility of the salt was determined only with difficulty as the solutions became very viscous and the limits of solubility very indistinct. Of the methods tried the most satisfactory was to add the dried perchlorate by increments to the mixed monomers under anhydrous conditions.

The salt was added until no further solution could be noted, and a slight amount of the salt remained undissolved. The solution was then warmed to complete solvation. The solubilities are shown in Table 1 (Ref. 4).

TABLE 1. Solubility of Potassium Perchlorate.

Organic binder solution			Mg(ClO ₄) ₂ weight, % in binder solution
Acrylonitrile weight, %	Butyl acrylate weight, %	Ethylene glycol weight, %	
100.0	0.0	0.0	38.0
0.0	100.0	0.0	42.2
45.0	45.0	10.0	48.8
72.5	22.5	5.0
22.5	72.5	5.0	43.8
50.0	50.0	0.0	47.4
48.3	48.3	3.4	46.6
46.7	46.7	6.6	47.6
81.6	16.7	1.7	39.0
16.7	81.6	1.7	39.7

To reduce viscosity of the solutions and to determine the solubility limit more closely, varying amounts of "inert" solvents such as ethanol or ethyl formate were added to the MgANBA system. After solution had occurred, the inert solvent was stripped off under vacuum. While more salt could be dissolved in this manner, the effect of the inert solvent on the curing process appeared to be deleterious.

The ideal diluent would reduce the viscosity, speed up the solution of the perchlorate, and be easily removed after solution was complete. The low boiling alcohols are not completely stripped off, and methanol at least, and under some conditions ethanol, are known to react with magnesium metal.

Ethyl formate was tested since a residual amount would not react with magnesium. However, monomer solutions containing ethyl formate diluent were found to polymerize to a significant degree as the magnesium perchlorate concentration reached about 35 to 40%. When this initial polymer was further polymerized in the sun, it gave a tenacious and quite rubbery product. Upon standing, the ethyl formate was apparently released and the polymer became more brittle, although it could not be stripped from the solution by the normal methods. When ethyl formate was used as a plasticizer (17% of the monomer system) the product of the polymerized solution with magnesium was brittle and generally had the same properties as those systems containing no plasticizer or low percentages of the ethylene glycol.

When acetone was used as the diluent, acetone and acrylonitrile were stripped off at about equal rates by distillation. Crystallization occurred when the magnesium perchlorate concentration was 48%. However, solution was again effected upon warming. The final concentration of monomers after stripping was about 34% butylacrylate and 17% acrylonitrile with the rest being residual acetone. The polymer derived from this mixture was quite flexible rather than hard and brittle as in most other systems. The volatility of the acetone would probably cause a definite aging effect on this system.

A run was made to determine whether or not a higher ratio of ethylene glycol would plasticize the polymer. A solution of 31% acrylonitrile, 33% butylacrylate and 36% ethylene glycol was prepared and, after dissolving 45% magnesium perchlorate, this gave a polymer as hard and brittle as before.

A summary of these special systems is given in Table 2 (Ref. 4).

TABLE 2. Special Binder Systems Made With Volatile Solvents.

Solution No.	Acrylonitrile, % ^a	Butyl acrylate, % ^a	Other, % ^a	Mg(ClO ₄) ₂ ^b solubility, parts by weight	Polymer properties
11	49.3	48.7	H ₂ O 2.0	40.4	Hard, brittle
12	41.6	41.1	Ethyl formate 17.3	46.6	Hard, brittle
13	23.7	23.3	Ethyl formate 50.3	45.8	Flexible, tenacious
14	17.3	33.9	Acetone 48.9	48.3	Flexible, tenacious
15	31.2	32.7	Ethylene glycol 36.1	44.6	Hard, brittle
16	49.8	50.2	Both as received	46.7	Hard, brittle

^aPercent by weight not including oxidizer content.

^bPercent by weight of total binder solution.

The only work on the volatile solvent diluted systems that appears anywhere near useful is that using acetone. Too much monomer is lost on stripping the solvent in the other cases. It also appears that controlling the organic monomer portion of the binder would be very difficult for routine mixing of pyrotechnic material. Work on this portion of the program was therefore suspended.

The magnesium perchlorate/acrylonitrile/butylacrylate system (MgANBA) was used throughout the development.

INITIAL MgANBA FORMULATIONS

Sensitivity. A typical mix containing magnesium perchlorate, butylacrylate-acrylonitrile, magnesium (gran 18) and sodium nitrate with benzoyl peroxide as catalyst was tested. Impact sensitivity was reported as one out of 20 fires at 120 cm with a 2.5 Kg weight. ABL sliding friction was found to be 236 pounds for the 50% point. Electrostatic testing showed 10 of 10 no fires at 0.25 joule. These data suggest that no undue hazards would be met with the finished mix.

Processing. All mixes were made in a 150 ml capacity Atlantic Research Corporation twin blade conical mixer at 50°C. The procedures in all were quite similar. The liquid binder material containing dissolved $Mg(ClO_4)_2$ was placed in the bowl. After the curing agent was added and mixed, sodium nitrate was added in two increments and thoroughly mixed. Next added was the magnesium in two increments. The mixture was then agitated for 15 minutes. Vacuum can be applied to the mixer if desired at anytime during mechanical mixing, but this was not done in this series. The completed mix was remotely dumped into a transfer container and then loaded into 1-inch-diameter molds with vibration applied during loading and for a short period afterward to promote settling and allow trapped gas to escape. After the mold was full, the material was cured at about 60°C until hard enough for combustion test. For most formulations, an overnight cure was adequate.

Experience gained with the initial cast-flare compositions indicated areas of potential improvement of properties such as fluidity, homogeneity, castability, and cure. The binder under consideration is highly hygroscopic and even traces of moisture might affect the cure and resulting physical properties. The sodium nitrate was dried before use to help alleviate this problem (Table 3).

Several mixes were then made with magnesium content of 50, 55, 60, and 65% to determine the effect of oxidizer to fuel ratio. Formulations with both granulation 17 and 18 magnesium were made to further compare the effect of fuel particle size. The data are presented in Table 4 and as can be seen suggest that the smaller particle size magnesium provides improved illumination efficiency, even though the burning rate is speeded

TABLE 3. Experimental Cast Formulations Composition and Properties.

Ingredients	Mix No.												
	6842	83611	83621	83651	83652	83661	83662	90141	90161	90162	90163	90211	90212
Magnesium, Z (Granulation)	56.0 (17)	60.0 (15)	60.0 (15)	60.0 (15)	60.0 (17)	60.0 (17)	60.0 (17)	14.42 (15)	60.0 (18)	60.0 (18)	60.0 (18)	60.0 (18)	60.0 (18)
Magnesium, Z (Granulation)								38.46 (17)					
Magnesium, Z (Granulation)								4.81 (18)					
Sodium Nitrate, Z (Particle Size)	37.0 (44u)	48.0 (150u)	20.0 (150u)	25.0 (150u)	20.0 (150u)	25.0 (150u)	28.0 (150u)	19.23 (150u)	28.0 (150u)	25.0 (150u)	20.0 (150u)	20.0 (150u)	25.0 (150u)
Viton A, Z	7.0												
Magnesium Perchlorate		6.0	10.0	7.5	10.0	7.5	6.0	11.54	6.0	7.5	10.0	0.5	0.5
Butylacrylate- acrylonitrile Formula: 50/50 Plus curing agent .02%		6.0	10.0	7.5	10.0	7.5	6.0	11.54	6.0	7.5	10.0	19.5	14.5
Cast Density, g/cc	1.62	1.561	1.572	1.485	1.549	1.423	1.556	1.305	1.256
Maximum theoretical density, g/cc	1.907	1.764	1.676	1.730	1.676	1.730	1.764	1.652	1.772	1.730	1.676	1.473	1.563
cp-sec/g $\times 10^3$	35.2	32.5	34.7	35.2	36.2	34.1	29.9	25.3	26.8	32.0	16.4	22.3	32.6
Burn rate, in/sec	0.106	0.466	0.421	0.0478	0.113	0.123	0.101	0.151	0.102

TABLE 4. Experimental 1.0-Inch-Diameter Cast Illuminating Formulations.

Ingredients	PL 6842 ^a Standard	90441	90442	90443	90581	90582	90583
Magnesium, % (Granulation)	56 (17)	55 (18)	50 (18)	65 (18)	55 (17)	60 (17)	65 (17)
Sodium nitrate, % (Particle Size)	37 (44 μ)	29 (Av. 110 μ)	34 (Av. 110 μ)	19 (Av. 110 μ)	29 (Av. 110 μ)	24 (Av. 110 μ)	19 (Av. 110 μ)
Viton A, %	7.0
Magnesium perchlorate dissolved, %		8	8	8	8	8	8
Butylacrylate acrylonitrile formula 50/50 plus curing agent		8	8	8	8	8	8
Cast density, g/cc		1.535	1.535	b	1.583	1.533	1.531
Theoretical maximum density, g/cc	1.907	1.739	1.719	1.699	1.739	1.719	1.699
cp, sec/g x 10 ³ ^c	32.0	29.3	33.5	24.0	27.5	42.2	39.5
Burn rate, in./sec ^c	0.108	0.047	0.046	b	0.150	0.110	0.140

^aPressed samples, 30 g. 7,500 psi.^bNot obtained.^cMeasurements are the average of four samples, all samples were in 0.030-inch-thick by 1.0-inch-OD stainless steel cases.

up considerably. One mix, 90582, was chosen for scale-up experiments. A more conventional pressed mix formulation, PL 6842, was burned during the test series for a comparison. Surprisingly, the cast mixes compared very favorably with PL 6842, which has been used for several years at NWC as the workhorse illumination mix.

Four mixes were made of this formula 90582, and cast into Micarta molds 1.75 inch diameter by 3 inches high. The data are shown in Table 5. Densities in three samples were close to 90% of theoretical, and burning rates for these were much less than for the original mix. One mix, 90711, showed a density of 88.5% of theoretical and burning rate was much faster. All four batches gave quite consistent results in cp sec/g.

TABLE 5. 1.75-Inch Diameter Flares of Cast Formulation 90582.

Ingredient	90582 ^a	90711 ^b	90712 ^b	90713 ^b	90714 ^b
Magnesium, % (granulation)	60.0 (17)	60.0 (17)	60.0 (17)	60.0 (17)	60.0 (17)
Sodium nitrate, % (particle size)	24.0 (110μ)	24.0 (110μ)	24.0 (110μ)	24.0 (110μ)	24.0 (110μ)
Magnesium perchlorate - fines %	8.0	8.0	8.0	8.0	8.0
Butylacrylate acrylonitrile formula 50/50 plus curing agent	8.0	8.0	8.0	8.0	8.0
Cast density, g/cc	1.533	1.522	1.541	1.547	1.544
Theoretical maximum density, g/cc	1.719	1.719	1.719	1.719	1.719
cp sec/g x 10 ³	42.2	41.6	46.4	38.8	44.0
Burn rate, in/sec	0.110	0.11	0.08	0.06	0.07

^a From results reported in Table 4, burned in 1-inch diameter stainless steel case.

^b Cast into 1.75-inch-diameter by 3-inch-high Micarta molds. About 250 grams of composition needed per mold, this in turn required preparation of four similar composition batches.

SCALE UP EXPERIMENTS

Because the heat releases for both dissolving the magnesium perchlorate and polymerization were not known, it was necessary to scale up mixing and casting by increments. The previous mixes were 250 grams each. The same formulation was now made in 600 gram batches. The efficiencies and burn times were not affected (Table 6) (Ref. 6). All mixing continued to be by remote control.

An attempt was made to improve the oxygen balance of the binder system by substituting methylacrylate for the butyl compound. The effect of sodium nitrate particle size was evaluated in the same experimental series of mixes. Up to this time "as received" sodium nitrate had been used. Now some mixes were prepared with reground and sieved sodium nitrate. The particle size distribution for the two types of sodium nitrate are given in Table 7 (Ref. 6) and the results upon burning the flares are given in Table 8 (Ref. 7). The data as to luminous efficiency and burn time are inconclusive. However, the volatility of the methylacrylate makes composition control difficult. Moreover, to control the fine particle size of the sodium nitrate, grinding and screening were necessary. The scale-up to a 5 pound batch was therefore made with butylacrylate and as-received sodium nitrate.

Larger batches with this binder system were made to obtain larger cast samples (3 inch diameter by 4 inches high). Metal content was set at 57, 60, and 63% with binder and perchlorate held at 8% in each formulation. The results (Table 9) (Ref. 8) are quite consistent with the earlier results with the smaller samples--efficiencies in the area of 43×10^3 cp sec/g and in burn time, about 9 seconds per inch.

Several mixes were made with the magnesium/sodium nitrate ratio 3:1, and one with the ratio 2:1. The material was cast into tubes 1.75 inches in diameter by 3 inches high, and also into cases 3 inches in diameter by 4 inches high. The limited data (Table 10) suggest a slightly higher efficiency for the larger samples (Ref. 8).

OXYGEN ENRICHMENT WITH NITRATED ORGANIC COMPOUNDS

Additional oxygen in a flare mix can be supplied by organic as well as inorganic compounds. Results of such work have been reported by other investigators. For comparison with the perchlorate-enriched system, mixes were made with triethylene glycol dinitrate (TEGDN). The binder used consisted of Dynamar HX 736, a prepolymer with a 30% oxygen content, and Dynamar HX 740 the curing agent (Ref. 9).

TABLE 6. Experimental Cast Formulations Compositions and Properties.

Batch No.	Composition	Weight, %	Burn rate, in/sec (average)	cp sec/gram $\times 10^3$	Sample weight, grams
DAS 91971	Mg powder (gran #17) NaNO ₃ (thru 60 mesh) retained on 140 mesh) Magnesium Perchlorate Butylacrylate/Acrylonitrile/ Ethylene Glycol (45/45/10) plus curing agent	60.0	0.13	Shot 1 42.0	146
		24.0		Shot 2 42.0	136
		8.0 8.0		Shot 3 42.0	129
DAS 92131	Mg powder (gran #17) NaNO ₃ (thru 140 mesh) retained on 200 mesh) Magnesium Perchlorate Butylacrylate/Acrylonitrile/ Ethylene Glycol (45/45/10) plus curing agent	60.0	0.12	Shot 1 42.2	134
		24.0		Shot 2 40.3	137
		8.0 8.0		Shot 3 42.9	131

TABLE 7. Particle Size Distribution.

Sodium nitrate (as received)		
US Standard screen size (mesh)	Micron size	Weight, % on
100	149
120	125	9.0
140	105	14.0
170	88	14.0
200	74	6.5
270	53	50.3
325	44	3.7
<325	44	2.5
Ground sodium nitrate		
270	53	0
325	44	100

TABLE 8. Experimental Cast Formulations Composition and Properties
(1.75 Inch Diameter x 3 Inch High Micarta Case).

Batch No.	Nominal composition	Weight, %	Burn rate, in/sec (average of 3)	cp sec/gram x 10 ³	Sample weight, grams
DAS 00161	Mg powder (gran 17) NaNO ₃ ground Magnesium perchlorate fines Methyl Acrylate/Acrylonitrile/ Ethylene Glycol (45/45/10)	60.0	6.1	Test 1 44.6	157
		24.0		Test 2 42.7	159
		8.0		Test 3 40.5	158
		8.0		Average 42.6	
DAS 00192	Mg powder (gran 17) NaNO ₃ as received Magnesium perchlorate fines Methyl Acrylate/Acrylonitrile/ Ethylene Glycol (45/45/10)	60.0	6.8	Test 1 46.3	161
		24.0		Test 2 49.5	161
		8.0		Test 3 57.9	160
		8.0		Average 51.2	
DAS 00193	Mg powder (gran 17) NaNO ₃ ground Magnesium perchlorate fines Butylacrylate/Acrylonitrile/ Ethylene Glycol (45/45/10)	60.0	7.0	Test 1 51.9	156
		24.0		Test 2 47.5	158
		8.0		Test 3 49.6	164
		8.0		Average 49.7	
DAS 00201	Mg powder (gran 17) NaNO ₃ as received Magnesium perchlorate fines Butylacrylate/Acrylonitrile/ Ethylene Glycol (45/45/10)	60.0	5.5	Test 1 50.8	156
		24.0		Test 2 46.0	157
		8.0		Test 3 47.0	152
		8.0		Average 47.9	

TABLE 9. Intensity of Cast Illumination Flares, Magnesium Perchlorate Reinforced Binder (3-Inch-Diameter).

DAS- batch	Composition	Weight, %	Sample No.	Sample weight, grams	cp sec/g $\times 10^3$	cp $\times 10^3$	Burn rate, in/sec
00441	Mg 17	57	1	688	41.9	795	9.3
	NaNO ₃	27	2	681	46.4	859	9.5
	Mg(ClO ₄) ₂	8					
	Binder	8					
00442	Mg 17	60	1	673	43.3	893	8.4
	NaNO ₃	24	2	675	47.5	1,090	7.8
	Mg(ClO ₄) ₂	8					
	Binder	8					
00443	Mg 17	63	1	665	42.0	770	9.2
	NaNO ₃	21	2	660	42.5	784	9.0
	Mg(ClO ₄) ₂	8					
	Binder	8					

NOTE: Mg 17--45/100 mesh
 NaNO₃--Davies, as received; see Table 7
 Binder--Butylacrylate, acrylonitrile, ethylene glycol,
 51/41/8 weight %
 Samples--3-inch diameter by 4 inches high, in cardboard
 flare cases.

TABLE 10. Intensity of Illumination Mixes With Nitrated Organic Binders.

DAS- batch	Composition	Weight, %	Sample No.	Sample weight, grams	cp sec/g $\times 10^3$	Average cp $\times 10^3$	Average burn rate, in/sec
00151 ^a	Mg 17	54	1	170	36.0	87.4	----
	NaNO ₃	27	2	173	31.4	82.3	----
	Binder	19	3	169	38.0	95.8	22.6
00221 ^a	Mg 17	60	1	159	38.9	131	----
	NaNO ₃	21	2	155	35.6	105	----
	Binder	19	3	159	42.3	120	17.3
00301 ^a	Mg ^b	62	1	177	31.8	128	----
	NaNO ₃	21	2	176	38.7	155	----
	Binder	17	3	177	41.8	211	13.6
00711 ^c	Mg 17	60	1	676	41.4	582	----
	NaNO ₃	21	2	684	39.9	550	----
	Binder	19	3	600	41.6	822	12.3
00712 ^c	Mg 17	62	1	690	45.7	528	----
	NaNO ₃	21	2	671	42.7	533	----
	Binder	17	3	600	40.9	633	14.3
00851 ^c	Mg 17	62		670	44.8	1,030	
	NaNO ₃ ^d	21.7					
	Binder	16.3					7.3
00852 ^d	Mg 17	62		681	42.6	763	9.5
	NaNO ₃	21.7					
	Binder	16.3					

NOTE. Magnesium, gran 17--45/100 mesh. NaNO₃--200/230 mesh.

Binder system--HX 736 resin 30 weight %, HX 740 curative
10 weight % with 60% of TEGDN added.

^aSamples--1.75-inch diameter by 3-inches high.

^bMagnesium 45/100 mesh 50%, 100/200 mesh 50%.

^cSamples--3-inch diameter by 4-inches high.

^dNaNO₃ Screen Analysis on 120 mesh 0.0% on 230 mesh 6.2%
on 140 mesh 10.0% on 270 mesh 10.6%
on 170 mesh 7.5% on 325 mesh 12.8%
on 200 mesh 39.0% through 325 mesh 13.8%

The binder and the TEGDN were mixed and the magnesium added with stirring. This reduced the dust hazard and somewhat "desensitized" the magnesium powder. The sodium nitrate was added by increments and after thorough blending, the mix was transferred to the flare cases. Cure was started at ambient temperature and completed at 140°F for 12 hours.

LARGE DIAMETER CANDLES

Both the magnesium perchlorate reinforced binder and the Dynamar-TEGDN system were used in larger batches (Table 11) to make possible castings of 4.25-inch and 7.25-inch diameters. The 4.25-inch flare cases were obtained from NAD/Crane, and they had been coated on the inner surface with a black pigment. The 7.25-inch cases were coated on the inner surface with Laminac 4116 and Lupersol DDM curing agent as had been done with the 3-inch cases previously prepared (Ref. 10 and 11). These cases were the same as for Mk 45 and Briteye flares.

The cases (both 4.25- and 7.25-inch sizes) were filled in a vacuum casting bell with an uninterrupted flow of material to prevent formation of voids, etc. Vacuum was continued after loading had been completed to reduce the possibility of entrapped air in the flare candle. It was not possible to vibrate the samples while they were under vacuum.

In general, candle cohesion was poor. During burn tests, Table 11, fragments fell from suspended candles or were ejected from flares in the upright position. In some instances there was very rapid disintegration, although all the pieces burned to completion. Apparently the candles shrank away from the case during curing. Only one candle remained in a suspended case (open end down) for the entire burn time. In a second instance, the candle burned in the case for 25 seconds, and some candle-power data were obtained from this portion of the flare. Both of these were made from the Dynamar formulation.

The data for one 4.25-inch-diameter candle with the Dynamar/TEGDN binder show an average candlepower of 2,130,000, with 88,500,000 total candlepower seconds. The data for a portion of a candle are not viable.

There was reason to suspect that case bonding was inadequate and the experiment was repeated. In this instance, the cases were coated on the inner surface with Laminac 4116 using Lupersol DDM as a curing agent. This was to serve as a bonding agent and adhered well to the tubes. While the coating was still uncured, the candle mix was loaded into the cases. The procedure was used for both the 4.25 inch and 7.25 inch diameter sizes, and for both the MgANBA and the Dynamar TEGDN binder systems. The candles were loaded with both vibration and vacuum to achieve a better packing density and to prevent voids. This had not been possible on the earlier experiments.

For comparison two Mk 45 flare candles (4.25 inch diameter) in the cardboard cases but without the aluminum sleeve burned with little fragmentation, and were not ejected from the cases. The total times were about 3 minutes and 15 seconds. The average intensity was 2.01×10^6 cp.

In previous experiments no meaningful intensity measurements could be obtained. In the present experiments, candle adherence to the cases was satisfactory. Two specimens with the magnesium perchlorate binder gave an average of 1.970×10^6 cp with an average efficiency of 62.3×10^3 cp sec/g. These were in the 4.25-inch cases (Table 12).

Apparently the larger flares (7.25-inch diameter) were lacking in cohesive strength. In the Dynamar binder system fragmentation occurred about 15-20 seconds after ignition, but the pieces continued to burn on the ground. In the magnesium perchlorate reinforced binder system violent fragmentation occurred very shortly after ignition. The larger fragments continued to burn on the ground.

X-ray examination of the larger casting (7.25-inch diameter) taken before firing showed that the castings with Dynamar binder system were less homogeneous than those with the perchlorate reinforced binder. Also there was greater separation between the casting and the shell case. Voids were also present in the Dynamar system that were not noticed in the perchlorate formulation.

Due to a requested change in emphasis of the Pyrotechnics Exploratory Development AirTask, no further work was done to improve the performance of large cast flares.

CONCLUSIONS

Castable flares with performance equivalent to that of conventionally prepared pressed flares are possible. Additional work to improve case bonding and optimize compositions and loading procedures are required, however, prior to incorporation in production devices. By the use of castable mixes, it should be possible to utilize noncylindrical flare cases and possibly further improve flare performance.

TABLE 11. Cast Illumination Flares, 4.25 and 7.7 Inches in Diameter.

Etch No.	Composition, weight %	Density, g/ml	Case No.	Burn time, total sec	Average candlepower 10^3	candlepower sec x 10^6	Notes
CT 120	Mg gr. 17 60	1.383	120-2				Cardboard cases Mk 45 diameter - 4.250 by 7.7 inches high
	NaNO ₃ 24	1.388	120-7				
	Binder #1 16	1.395	120-8		Case inhibitor failure		
		1.384	120-10		Candle ejected		
CT 120A	Mg gr. 17 60	1.525	120-21				Cardboard case Briteye diameter - 7.250 by 13.5 inches high
	NaNO ₃ 24						
	Binder #1 16						
CT 121	Mg gr. 17 58.1	1.344	121-1				Mk 45 diameter 7.3 inches high
	NaNO ₃ 23.2						
	Cab-O-Sil 0.7						
	Binder #1 18.0						
CT 122	Mg gr. 17 62	1.396	122-5	25	2.97	73.1	Mk 45 cases
	NaNO ₃ 21	1.326	122-6	..	Candle ejected	
	Binder #2 17	1.405	122-9	41	2.13	88.5	
		1.348	122-12	..	Candle ejected	
CT 122A	Mg gr. 17 62	1.551	122-20	..	Candle ejected	Briteye case
	NaNO ₃ 21						
	Binder #2 17						

NOTE: Binder #1 - [Mg(ClO₄)₂/butylacrylate/acrylonitrile/ethylene glycol (100/51/41/8 parts by weight)], 1 part curing agent.

Binder #2 - Dynamar resin HX 736/TEGDN/Dynamar curative (30/60/10 parts by weight).

TABLE 12. Luminous Intensity of Cast Illumination Flares, 4.25 Inch Diameter.

Batch No.	Composition, weight %	Average intensity, cp x 10 ⁶	Efficiency cp sec/g x 10 ³	Burn time, sec	Burn rate, sec/in	Density, g/cc	Candle weight, grams
120C	Mg gran 17 NaNO ₃ , as received	59.4 23.8	62.4	75	8.1	1.464	3,147
	MgClO ₄	7.9					
	Binder ^a	8.9					
120D	Mg gran 17 NaNO ₃ , as received	59.4 23.8	62.3	83	9.3	1.538	3,172
	MgClO ₄	7.9					
	Binder ^a	8.9					
122B	Mg gran 17 NaNO ₃ Davies Binder ^b	60 21.0 19.0	56.3	83	9.7	1.619	3,200
122C	Mg gran 17 NaNO ₃ Davies Binder ^b	60 21.0 19.0	47.1	75	8.1	1.495	3,195

^aAcrylonitrile/butylacrylate/ethylene glycol/50 9/40. /8.8 weight %.^bPrepolymer resin HX 730 TFGDN/curing agent HX 740 30/60/10 weight %.

REFERENCES

1. Air Force Armament Laboratory. Binder Study - Visual Cast Flare (U), by H. R. Waite and Y. Arikawa. Eglin Air Force Base, Crane, Indiana, March 1967. (RDTR No. 92, publication UNCLASSIFIED.)
2. Dow Chemical Company. Monthly Report under Contract No. F08635-71-C-0120 for the Air Force, dated 4 June 1971.
3. -----. Monthly Report under Contract No. F08635-71-C-0120 for the Air Force, dated 28 July 1971.
4. Naval Weapons Center. Pyrotechnic Research and Development, July-September 1968 (U). China Lake, Calif., NWC, September 1968. (NWC TP 4641, publication CONFIDENTIAL.)
5. -----. Pyrotechnic Research and Development, January-March 1969 (U). China Lake, Calif., NWC, March 1969. (NWC TP 4731, publication CONFIDENTIAL.)
6. -----. Pyrotechnic Research and Development, April-June 1969 (U). China Lake, Calif., NWC, June 1969. (NWC TP 4763, publication CONFIDENTIAL.)
7. -----. Pyrotechnic Research and Development, January-March 1970 (U). China Lake, Calif., NWC, March 1970. (NWC TP 4915, publication CONFIDENTIAL.)
8. -----. Pyrotechnic Research and Development, April-June 1970 (U). China Lake, Calif., NWC, June 1970. (NWC TP 4988, publication CONFIDENTIAL.)
9. Picatinny Arsenal. Parameters Affecting Performance Characteristics of Cast Flare Systems (U), by Stephen C. Dollman, et al. Dover, N. J. (Unnumbered Report pages 693 to 711, publication UNCLASSIFIED.)
10. Naval Weapons Center. Pyrotechnic Research and Development, October-December 1970 (U). China Lake, Calif., NWC, December 1970. (NWC TP 5083, publication CONFIDENTIAL.)
11. U. S. Naval Ammunition Depot. Four Cast Pyrotechnic Masses (U), by B. E. Douda. NAD/Crane, 5 May 1963. (RDTN No. 35, publication CONFIDENTIAL.)